BB

PATENT SPECIFICATION

NO DRAWINGS

848,230

Date of Application and filing Complete Specification: Aug. 16, 1957. *No. 25931/57.*

Application made in France on Aug. 27, 1956.

Complete Specification Published: Sept. 14, 1960.

Index at acceptance :—Class 1(3), A1D1, A1G50D1, A1N(4A2A:20:34)

International Classification :—C01f,g.

COMPLETE SPECIFICATION

A Process of Separately Recovering Aluminium, Iron and Titanium Values from Material such as " Red Mud " containing said Values

We, SOCIETE DES BLANCS DE ZINC DE LA MEDITERRANEE Eug. CHABAURY-PIERRE GINDRE & CIE, a French Body Corporate of 137, Pont-de-Vivaux—Marseille—(Bouches du Rhone), France, do hereby declare the invention, for which we pray that a patent may be granted to us and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to a process of separately recovering aluminium, iron and titanium values from material containing said values, such as " red mud " obtained during the manufacture of aluminium from bauxite.

There exist for industrial purposes a number of products which have large contents of alumina and contain furthermore valuable components the extraction of which is of considerable interest.

Among these, may be mentioned the so-called " red mud " which forms the residual material obtained in the production of aluminium from bauxite by the conventional methods and which has the following average composition:

Alumina	: 25 to 30%
Iron oxides	: 25 to 35%
Lime	: 10 to 15%
Silica	: 5 to 10%
Titanium oxide	: 3 to 6%

Similarly, the residual material obtained in the manufacture of ferro-titanium, starting from rutile or ilmenite, generally contains the following components:

Alumina	: 25 to 30%
Titanium oxide	: 10 to 25%
Iron oxides	: 10 to 15%

The treatment of such residual materials is difficult, so that it is not generally used, since the conventional alkaline smelting treatments are expensive and lead often only to an incomplete and difficult recovery of the components.

[*Price 3/6*]

The process according to the present invention allows this recovery in an economical manner and on an industrial scale through an easy separation of the valuable products contained in such residual materials and through a transformation thereof into pure products which may be readily used or into concentrates which are easily treated.

According to the present invention, there is provided a process of separately recovering aluminium, iron and titanium values from material containing said values, which process comprises subjecting said material to an alkaline sintering with sodium carbonate at a temperature of from 800-850°C. for a duration of from 10-30 minutes and in the presence of from 1-2% by weight, based on the weight of alumina in the material, of sodium borate as catalyst, washing the resulting sintered product with boiling water to obtain an insoluble residue containing iron and titanium in insoluble form, and an alkaline solution containing salts of metals the oxides of which are soluble in sodium hydroxide, reducing the insoluble residue by smelting to obtain a fused mass of iron and a slag containing titanium substantially free from iron, and decomposing the alkaline solution with carbon dioxide to precipitate alumina.

The amount of sodium carbonate employed in the alkaline sintering varies according to the aluminium content of the raw material which is being treated.

The alkaline sintering removes about 95% of the alumina existing in the raw material with an expenditure of heat which is comparatively small since the temperature of sintering is not very high and the duration of the reaction is very short.

As disclosed hereinafter, the sodium carbonate used in the alkaline sintering may be recovered to the extent of at least 90%.

The washing of the sintered product produced in the first stage of the treatment

Price 2/6

provides two separate products, namely an insoluble residue containing iron in the form of iron oxides, titania, and oxides of other elements such as alkaline earth oxides, magnesia and silica, and an alkaline solution which contains together with the excess sodium carbonate, salts such as, for example, aluminates, chromates, vanadates and manganates.

The third stage of the treatment, in which the aforesaid insoluble residue is smelted, may be performed in an electric furnace, for example in the manner described and claimed in Patent Specification No. 802,336. By the smelting treatment there is obtained a cast iron of good commercial grade and a slag containing titanium substantially free from iron. This slag contains, according to the raw material used, various components of interest, chiefly titanium oxide, the extraction of which may be performed through conventional methods, such as in particular the treating methods disclosed and claimed in Patent Specifications Nos. 789,104 and 791,302.

In the fourth stage of the treatment, the alkaline solution formed in the second stage of the treatment is treated with a stream of carbon dioxide gas from an external source or, more simply, a stream of furnace fumes containing a large proportion of carbon dioxide, which stream is passed through the filtered solution so as to precipitate hydrated alumina, which may then be treated after separation by conventional methods.

The solution obtained after the separation of the alumina may be treated to recover, for example, manganese chromium and vanadium by suitable well known methods. Alternatively, the solution may be concentrated and dried by atomization or the like methods to obtain sodium carbonate which is somewhat coloured by the above metals but which can however be used in the alkaline sintering stage.

The process of the present invention may also be used for aluminium ores which have not undergone any treatment. In particular, this may be the case for bauxites, the compositions of which are somewhat similar to the compositions given hereinabove with merely higher contents of alumina (40 to 65%).

For a better understanding of the invention and to show how the same may be carried into effect, the following illustrative examples will now be given.

EXAMPLE 1

Treatment of red mud

The red mud used had the following composition:

27% Al_2O_3
31.24% Fe_2O_3
41.76% SiO_2, TiO_2, CaO, Na_2O, Cr_2O_3

100 kg. of the red mud admixed intimately with 93 kg. of anhydrous sodium carbonate and 0.500 kg. of sodium borate were heated at 820°C. for 30 minutes.

The sintered product obtained which was very crumbly was finely crushed before it was washed with boiling water. It was then filtered so as to separate the filtrate from the residue. Furnace fumes containing a large proportion of carbon dioxide were bubbled through the filtrate and an abundant precipitate of hydrated alumina was obtained, which precipitate was collected and then roasted to produce 25.7 kg. of alumina (Al_2O_3) which was very white. The yield of alumina was 95%.

Through concentration and atomization of the remaining filtrate 84 kg. of sodium carbonate was recovered (90% of the sodium carbonate used in the procedure was thus recovered).

Analysis of the aforementioned residue showed that the latter contained 54% of iron oxide (Fe_2O_3); this residue could therefore be considered as an iron ore similar to the lime-containing low grade iron ore obtained from Lorraine and was treated in a similar manner by a reduction smelting operation in an electric furnace to produce a high grade cast iron and a slag containing titanium substantially free from iron.

EXAMPLE 2

Treatment of a residue obtained in the production of ferro-titanium

The residue used had the following composition:

29.62% Al_2O_3
10.38% Fe (expressed as Fe_2O_3)
22.12% TiO_2
38% SiO_2, MgO, MnO, CaO, oxides of vanadium and chromium.

100 kg. of the residue was admixed intimately with 102 kg. of anhydrous sodium carbonate and 0.300 kg. of sodium borate and heated at 820°C. for 30 minutes.

After operating in the manner described in Example 1, there were obtained 28.1 kg. of very white alumina (Al_2O_3). The yield of alumina was 94.9% of theory.

The residual liquid, which had an intense deep green colour, contained manganese. The latter was precipitated in the form of manganese oxide MnO_2 to obtain 3.7 kg. of MnO_2.

The resulting colourless alkaline solution was concentrated and atomized so as to obtain 92.5 kg. of anhydrous sodium carbonate which corresponds to a recovery of 90.6% of the sodium carbonate used in the alkaline fusion treatment.

There was also obtained by operating in the manner described in Example 1 a residue containing 19.2% of Fe (expressed as Fe_2O_3) and 40.96% of TiO_2. This residue may be considered as a titanium ore i.e. a concentrate of TiO_2, and it may be treated as such for the extraction of TiO_2.

EXAMPLE 3

Treatment of bauxite

The composition of the bauxite was as follows:

61.5% Al_2O_3
2.5% SiO_2
2.8% TiO_2
13% Fe_2O_3
20.2% losses in roasting.

100 kg. of the above bauxite were admixed intimately with 212 kg. of anhydrous sodium carbonate and 1 kg. of sodium borate and the mixture heated at 820°C. for 30 minutes.

By operating in a similar fashion to that described in Example 1, there were obtained 58.5 kg. of very white alumina (Al_2O_3) so that the yield of extracted alumina was about 95.1%. After concentration and atomization of the remaining liquid, there were obtained 190.5 kg. of anhydrous sodium carbonate which corresponds to a recovery of 89.8% of Na_2CO_3 used in the process.

There was also obtained a residue containing 80% of Fe_2O_3.

As in the case of Example 1, the residue may be considered as a true iron ore similar to roasted pyrites and it may be treated in the same manner to obtain cast iron.

WHAT WE CLAIM IS:—

1. A process separately recovering aluminium, iron and titanium values from material containing said values, which process comprises subjecting said material to an alkaline sintering with sodium carbonate at a temperature of from 800-850°C. for a duration of from 10-30 minutes and in the presence of from 1-2% by weight, based on the weight of alumina in the material, of sodium borate as catalyst, washing the resulting sintered product with boiling water to obtain an insoluble residue containing iron and titanium in insoluble form, and an alkaline solution containing salts of metals the oxides of which are soluble in sodium hydroxide, reducing the insoluble residue by smelting to obtain a fused mass of iron and a slag containing titanium substantially free from iron, and decomposing the alkaline solution with carbon dioxide to precipitate alumina.

2. A method according to Claim 1, wherein the filtrate obtained by filtration of the alumina precipitate is concentrated and dried to obtain sodium carbonate which is used in the alkaline sintering step.

3. A process of separately recovering aluminium, iron and titanium values from material containing said values, substantially as described in any one of the foregoing specific Examples.

HASELTINE, LAKE & CO.,
28, Southampton Buildings,
London, W.C.2,
Agents for the Applicants.

Printed for Her Majesty's Stationery Office by Wickes & Andrews, Ltd., E.C.4. 684/2.—1960.
Published at The Patent Office, 25, Southampton Buildings, London, W.C.2, from which copies may be obtained.